

NATIONAL BUREAU OF STANDARDS REPORT

10 065

Progress Report
on
CHARACTERIZATION OF A COMPOSITE RESIN-SILICA
MATERIAL DEVELOPED FOR TEMPORARY POSTERIOR RESTORATIONS



U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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CHARACTERIZATION OF A COMPOSITE RESIN-SILICA MATERIAL DEVELOPED FOR TEMPORARY POSTERIOR RESTORATIONS

by

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Abstract

The objective of this investigation was to characterize a resin-silica composite cement developed for use as a temporary posterior restorative material. The material is based on a monomer formulation of a ternary eutectic of isomeric phthalate esters of 2-hydroxyethyl methacrylate, and a reinforcing filler of vitreous silica and an X-ray opaque glass. The following properties were evaluated: setting time, temperature rise on setting, setting shrinkage, tensile and compressive strength, elastic moduli (Young's and shear), thermal expansion, indentation resistance, hardness, water sorption, solubility, color stability and optical opacity.

1. Introduction

A resin silica composite cement, developed for use as a temporary posterior restorative material was evaluated with respect to its mechanical and physical properties. The material is based on formulations previously developed at the National Bureau of Standards for use in anterior restorations.^{1,2}

The monomer is essentially a liquid ternary eutectic of isomeric phthalate esters of 2-hydroxy ethyl methacrylate.³ The reinforcing filler consists of irregular particles of vitreous silica and an x-ray opaque barium fluoride glass, both of which were treated with a vinyl silane coupling agent.¹ Benzoyl and lauroyl peroxides were added as accelerators. The treated powder was sifted through a No. 100 United States Standard sieve. It is desired that this material withstand the forces of mastication and abrasion for at least two years; be radio opaque so that it is possible to distinguish between the material and remaining or recurring caries and that it exhibit low pulpal irritation. Esthetic properties are of secondary importance, since the material is intended for temporary posterior restorations.

2. Experimental procedures

All test specimens were prepared at $23 \pm 2^{\circ}\text{C}$ and $50 \pm 10\%$ relative humidity. Samples to be tested dry were stored

in a 37°C air bath, and those to be tested in the wet condition were stored in distilled water at 37°C from the time of removal from the mold until time of test. Three mixing consistencies, 1.10, 1.35 and 1.45 gm of powder, incorporated into 0.4 ml of liquid, were used throughout this study. The 1.35 gm mix was considered by the operators to have an optimum working consistency. Mixing was accomplished by incorporating the entire amount of powder into the liquid at one time and spatulating for one minute and 15 seconds on a glass mixing slab.

2.1 Setting time

Setting time was determined by the method outlined in American Dental Association specification no. 9 for dental silicate cement⁴ with one modification. Instead of using a single ring to contain the material, a Teflon mold, approximately 100 mm long x 50 mm wide x 6 mm thick, having six cylindrical cavities approximately 6 mm in diameter, was used. This mold was placed on a glass plate, filled from a single mix of material, and each cylinder was covered with a small strip of foil.

2.2 Temperature rise on setting

Temperature rise on setting was determined by the method described by Walcott and others⁵ using a mold which produced a specimen of approximately 200 mm³. The increase in

temperature above room temperature was measured, rather than above 37°C, because the increase is small and occurs relatively quickly.

2.3 Setting shrinkage

Setting shrinkage values were obtained interferometrically in much the same manner as described in American Dental Association specification No. 1 for alloy for dental amalgam.⁴ A 5 x 10 mm mold, preheated to 37°C was filled with the material and transferred to a 37°C oven. Four minutes from the beginning of mix the specimen was removed from the mold, positioned on the dental interferometer and a fiducial reading was taken at five minutes. Additional readings were made each minute during the first fifteen minutes, at varying intervals thereafter, and a final reading was made at 24 hours.

2.4 Compressive strength

Six by twelve millimeter cylinders for compressive strength determinations were prepared as described in American Dental Association specification No. 9 for silicate cement.⁴ These specimens were crushed using a head speed of 0.5 mm/min. Strength tests were made on specimens which had been stored wet for 6 hours, 1, 7, and 28 days, and on specimens which had been stored dry for 90 min, 1 day and

28 days. All time intervals were measured from beginning of mix. Six specimens were prepared for each testing time.

2.5 Tensile strength

Specimen preparation size and time of test were identical to that for compressive strength. Tensile values were determined by the diametral compression method using a head speed of 51. mm/min. In order to record the rapid change in load, a Visicorder oscillograph was attached to the testing machine.

2.6 Elastic moduli

Young's modulus was determined by two different methods. The first method employed Tuckerman optical strain gages, with a one quarter inch gage length, attached to dumb bell shaped specimens having a straight center portion approximately 2.5 x 2.5 x 20 mm. The specimen was tested using a head speed of 0.13 mm/min. Strain gage readings were made at 5 pound load intervals and the stress-strain curves plotted. Six specimens of each consistency were tested.

An ultrasonic technique⁶ also was used to determine both the Young's and shear modulus of the material. Cylinders, 10 mm in diameter by 6 mm long, were tested.

Additional shear modulus values were obtained using the torsion pendulum⁷ method. Specimens, 2.5 mm in diameter and 20 mm long were tested.

All specimens used for moduli determinations were at least one week old.

2.7 Coefficient of thermal expansion

The coefficient of thermal expansion for these materials was determined interferometrically.⁸ The values were obtained from two heating and two cooling runs for specimens 4 mm in diameter x 8 mm in length tested wet.

2.8 Indentation resistance

The depth of penetration and recovery of the depth of indentation were obtained by the method outlined in American Dental specification No. 15 for acrylic resin teeth⁴ with the exception of specimen size. In this study, the specimens were discs twenty mm in diameter and 2 mm thick. Determinations were made on specimens stored in water for 1 1/2 and 6 hours, 1, 7 and 28 days, and on specimens stored dry for 1 and 28 days. Three determinations on each of three specimens (total of 9 determinations) were made at all time intervals.

2.9 Hardness

Hardness values were obtained using the Rockwell Superficial Hardness tester on the same type of specimen and at the same time intervals as for indentation resistance. Five determinations on each of three specimens (total of 15 readings) were made at all time intervals. The Rockwell 30 W scale, thirty kilogram major load with a 3.2 mm diameter ball indenter, was used. The major load remained in contact with the specimen for 15 seconds and the dial reading was recorded 15 seconds after removal of the major load.

2.10 Water sorption

Water sorption was determined by the method described in American Dental Association specification No. 12 for denture base polymers with the exception of specimen size. In this study discs 20 mm in diameter by 2 mm thick were used. In addition, after the 24 hours determination, the specimens were replaced in distilled water and reweighed at 24 hours intervals until constant.

2.11 Solubility

Solubility values were obtained by the method outlined in American Dental Association specification No. 9 for

silicate cements.⁴ One set of specimens was removed from distilled water after 24 hours immersion, and another set was allowed to remain in distilled water for 7 days.

2.12 Color stability

Twenty by two millimeter discs of these materials were exposed to the radiation of a lamp as described in American Dental Association specification No. 12 for denture base polymers⁴ for 24 hours and 7 days. Duplicate tests were made for each ratio and time.

2.13 Optical opacity

The optical opacity of these materials were determined by the method outlined in American Dental Association specification No. 9 for silicate cements.⁴

2.14 X-ray opacity

Radiographs of teeth containing restorations made from the composite material were made employing standard dental procedures.

3. Results

All values reported are averages and the coefficient of variation was 10% or less in every case.

3.1 Setting time

A setting time of five minutes, as measured from beginning to mix, was found for all three consistencies.

3.2 Temperature rise on setting

Results of temperature rise on setting are given in Table 1. Temperature rise ranged from 5° to 7°C above room temperature and occurred from four to 5 1/2 minutes from beginning of mix.

3.3 Setting shrinkage

Typical curves for the setting shrinkage are given in Figure 1. The initial change was very rapid and approximately 50% of the shrinkage occurred within the first fifteen minutes. The twenty-four hour value for the two higher consistencies was 0.4% and that for the 1.10 gm mix was 0.5%.

3.4 Compressive strength

Compressive strength data, Table 2, for specimens tested wet, show that the powder-liquid ratio did not significantly affect this property. However, each consistency did show an increase in strength with time. Six hour strengths were on the order of 165 MN/m² (24,000 psi) and increased to about 196 MN/m² (28,000 psi) after one week. These data indicate strengths which are about 1/2 that of amalgam, comparable to silicates and twice that of acrylic resins. Storing the specimens dry, Table 2, had

little or no effect on the compressive strength of these materials at 24 hours, but dry specimens did tend to be slightly stronger at 28 days.

3.5 Tensile strength

The early tensile strength of the thinnest mix, Table 3, is lower than that of the other two consistencies, 26 MN/m^2 (3700 psi) as compared to 30 MN/m^2 (4400 psi). There is a significant increase from the 6 hour to the 7 day and 28 day strength for all ratios. As in the case of compressive strength, storage conditions did not have a significant effect on the tensile strength values.

3.6 Elastic moduli

As can be seen from Table 4, there is little variation in Young's modulus with the consistency of the mix. Variations in results by the two different methods is much greater. The optical strain gage values presented in the table were calculated up to stresses of 35 MN/m^2 (5,000 psi). If only the data below 14 MN/m^2 (2,000 psi) stress are used the results of the two methods are much closer; approximately $14 \times 10^3 \text{ MN/m}^2$ (2.0×10^6 psi) for the strain gage method as compared to $172 \times 10^3 \text{ MN/m}^2$ (2.6×10^6 psi) for the ultrasonic technique.

Shear modulus is not significantly effected by the stiffness of the mix, nor does the method used give great variation. The torsion pendulum method value is approximately $6.5 \times 10^3 \text{ MN/m}^2$ ($0.8 \times 10^6 \text{ psi}$) as compared to $6.9 \times 10^3 \text{ MN/m}^2$ ($1.10 \times 10^6 \text{ psi}$) for the ultrasonic.

3.7 Coefficient of thermal expansion

Table 5 presents the values obtained for the coefficient of thermal expansion. As can be seen, the value is dependent upon the temperature range involved; all three consistencies having smaller coefficients at the lower end of the temperature range. The 1.35 gm mix is consistently lower than the other two consistencies, 30 ppm over the entire range as compared to 39 ppm for the 1.10 and 34 ppm for the 1.45gm mix. A possible explanation may be that the thinnest mix, 1.10 gm powder / 0.4 ml monomer contains less of the silica filler. The 1.45 gm mix is quite stiff, which makes it difficult to insure a homogeneous specimen. These coefficients of thermal expansion are approximately 1/4 to 1/3 that of the acrylics and about 10 ppm higher than amalgam.

3.8 Indentation resistance

The powder liquid ratio does not have a significant effect on the indentation resistance of these materials, Table 6.

They do exhibit a decrease in depth of penetration and an increase in percent recovery on aging from 90 minutes to 28 days. Values for both properties of specimens stored dry were within a few percent of the values for specimens stored wet.

3.9 Hardness

There appears to be a slight difference in the hardness of the thin mix and the heavier ones at early times, Table 7, but this difference decreases with time and could not be considered significant after seven days. Specimens stored dry gave values within 2 hardness numbers of the values obtained on the wet specimens. This is within the reproducibility of the test method.

3.10 Water sorption

Water sorption, Table 8, after 24 hours is 0.2 to 0.3 mg/cm². Continuing the soaking for seven days increased the value to 0.4 mg/cm² with no significant difference in the values for the three consistencies.

3.11 Solubility

Solubility of this material is low when compared to silicate cements, Table 9. The specification limit for solubility of silicate cements is 1.0% after 24 hours.

Immersing the composite resin in distilled water for 24 hours gave a solubility of 0.15%. After 7 days immersion, the 1.10 and 1.45 gm mixes increased to 0.22%, but the 1.35 gm mix remained unchanged.

3.12 Color stability

Color stability of these materials is good. Even after 7 days exposure to the lamp, there was no perceptible color change.

3.13 Optical opacity

This material is somewhat more opaque than silicates. All three consistencies were slightly higher, between 0.55 and 0.60, than the 0.55 Co₇₀ contrast ratio allowed for silicate cements.

3.14 X-ray opacity

Figure 2 illustrates the x-ray opacity of these cements. The restorations in the distal and mesial of the left central are made from a composite resin which did not contain a radio opaque glass. Those in the mesial and distal of the right central are made from the material under investigation.

4. Discussion

The data reported indicates that accurate proportioning is not a critical factor for optimizing the properties

evaluated. The five minute setting time provides sufficient working time for normal clinical use. Considering the size of the specimen, approximately 200 mm³, a temperature rise of 5° to 7°C would not be considered sufficient to injure the pulp. While the compressive strength of these materials is similar to that of silicate cements, the tensile strength is 5 to 10 times the reported values for silicates and about 3/4 the value reported for amalgam. The water sorption and solubility are low as compared to acrylics and silicate cements respectively. Although these composites are somewhat more opaque than silicates or natural tooth structure, this is not a great disadvantage since the material is intended for temporary posterior restorations. A significant advantage of this material is its radio-opacity, which permits delineation of the cement and remaining or recurring caries.

5. Conclusions

On the basis of the physical and mechanical properties reported, it appears that this composite resin cement has potential as a temporary posterior restorative material. Since no criteria are available to determine from laboratory data alone, whether or not a material will be successful

in use, clinical research and pulpal response tests, are now necessary to ascertain the suitability of the material for its intended purpose.

Table 1
Temperature Rise on Setting

| P : L* | Temp.** Rise °C | Peak Time min. |
|--------|-----------------------|----------------------|
| 1.10 | 6.7 | 4.0 |
| 1.35 | 4.9 | 5.5 |
| 1.45 | 5.1 | 5.5 |

* Gm. powder in a 4 ml monomer

** °C above room temperature

Table 2
Compressive Strength

| Age | P:L* | 1.10 | | 1.35 | | 1.45 | |
|-----------------------|------|--------|-------------------|--------|-------------------|--------|-------------------|
| | | PSI | MN/m ² | PSI | MN/m ² | PSI | MN/m ² |
| 6 hrs.-wet** | | 24,500 | 169 | 24,400 | 168 | 23,600 | 163 |
| 24 hrs.-wet dry*** | | 26,600 | 183 | 26,200 | 181 | 25,700 | 177 |
| | | 26,400 | 182 | 26,000 | 179 | 26,700 | 184 |
| 7 days-wet | | 28,600 | 197 | 28,500 | 196 | 29,200 | 201 |
| 28 days-wet dry | | 27,200 | 188 | 27,600 | 190 | 27,500 | 190 |
| | | 28,500 | 196 | 31,200 | 215 | 29,200 | 201 |

* Gm powder in 0.4 ml monomer

** Specimens stored in distilled water at 37°C

*** Specimens stored in 37°C air bath

Table 3
Tensile Strength

| Age | P:L* | 1.10 | | 1.35 | | 1.45 | |
|--------------|------|-------|-------------------|-------|-------------------|-------|-------------------|
| | | PSI | MN/m ² | PSI | MN/m ² | PSI | MN/m ² |
| 6 hrs.-wet** | | 3,700 | 26 | 4,400 | 30 | 4,400 | 30 |
| 24 hrs.-wet | | 4,700 | 32 | 5,300 | 36 | 4,800 | 33 |
| dry*** | | 4,800 | 33 | 4,600 | 32 | 5,200 | 36 |
| 7 days-wet | | 5,900 | 41 | 5,900 | 41 | 5,500 | 38 |
| 28 days-wet | | 5,200 | 36 | 5,700 | 39 | 6,000 | 41 |
| dry | | 5,400 | 37 | 5,300 | 36 | 5,600 | 39 |

* Gm powder in 0.4 ml monomer

** Specimens stored in distilled water at 37°C

*** Specimens stored in 37°C air bath

Table 4

Elastic Moduli

| Modulus \ P:L* | 1.10 | | 1.35 | | 1.45 | |
|------------------------------|-------------------|-------------------|-------------------|-------------------|-------------------|-------------------|
| | PSI | MN/M ² | PSI | MN/M ² | PSI | MN/M ² |
| Young's | 1.2×10^6 | 8.3×10^3 | 1.3×10^6 | 9.0×10^3 | 1.4×10^6 | 9.6×10^3 |
| Ultrasonic | 2.5 | 17 | 2.6 | 18 | 2.4 | 16 |
| Shear Torsion Pendulum | 0.7×10^6 | 4.8×10^3 | 0.8×10^6 | 5.5×10^3 | 0.8×10^6 | 5.5×10^3 |
| Ultrasonic | 1.0 | 6.9 | 1.0 | 6.9 | 0.9 | 6.2 |

* Gm powder in 0.4 ml monomer

Table 5

Thermal Expansion

| <u>P:L*</u> | <u>24-42°C</u> <u>PPM/°C</u> | <u>42-58°C</u> <u>PPM/°C</u> | <u>24-58°C</u> <u>PPM/°C</u> |
|-------------|---------------------------------|---------------------------------|---------------------------------|
| 1.10 | 37 | 41 | 39 |
| 1.35 | 28 | 32 | 30 |
| 1.45 | 31 | 38 | 34 |

* Gm powder in 0.4 ml monomer

Table 6

Indentation Resistance
(Wet Storage)

| Age \ P:L* | 1.10 | | 1.35 | | 1.45 | |
|------------|-------------------------------|--------------------|-------------------------------|--------------------|-------------------------------|--------------------|
| | depth of penetration mm | Recov- ery % | depth of penetration mm | Recov- ery % | depth of penetration mm | Recov- ery % |
| 90 min. | 0.08 | 54 | 0.07 | 56 | 0.07 | 58 |
| 6 hrs. | .07 | 64 | .07 | 63 | .07 | 67 |
| 1 day | .06 | 73 | .07 | 69 | .06 | 70 |
| 7 days | .06 | 73 | .06 | 74 | .05 | 74 |
| 28 days | .05 | 74 | .06 | 77 | .05 | 78 |

* Gm powder in 0.4 ml of monomer

Table 7

Hardness

| P:L* | 1.10 | 1.35 | 1.45 |
|------------|-------------|-------------|-------------|
| <u>Age</u> | <u>30 W</u> | <u>30 W</u> | <u>30 W</u> |
| 90 min. | 42 | 50 | 51 |
| 6 hrs. | 52 | 57 | 59 |
| 1 day | 61 | 65 | 64 |
| 7 days | 68 | 71 | 72 |
| 28 days | 69 | 72 | 72 |

* Gm powder in 0.4 ml of monomer

Table 8

Water Sorption

| <u>P:L*</u> <u>Age</u> | <u>1.10</u> <u>mg/cm²</u> | <u>1.35</u> <u>mg/cm²</u> | <u>1.45</u> <u>mg/cm²</u> |
|---------------------------|---|---|---|
| 24 hrs. | 0.25 | 0.26 | 0.16 |
| 6 days | 0.40 | 0.39 | 0.33 |
| 7 days | 0.42 | 0.39 | 0.35 |

* Gm powder in 0.4 ml monomer

Table 9

Solubility

| <u>P:L*</u> <u>Age</u> | <u>1.10</u> <u>%</u> | <u>1.35</u> <u>%</u> | <u>1.45</u> <u>%</u> |
|---------------------------|-------------------------|-------------------------|-------------------------|
| 24 hrs. | 0.16 | 0.15 | 0.16 |
| 7 days | 0.22 | 0.15 | 0.22 |

* Gm powder in 0.4 ml monomer

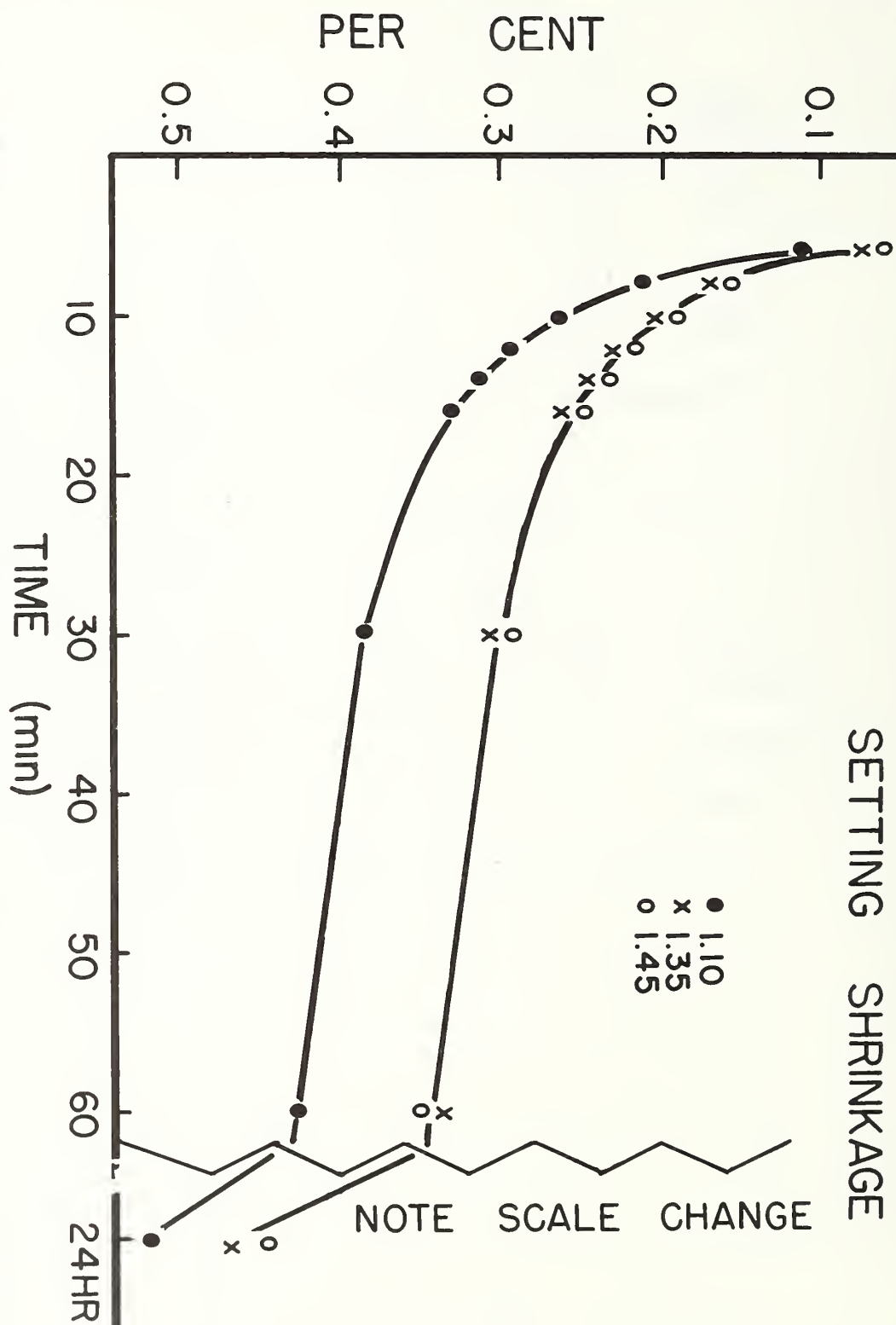


Fig. 1. Setting Shrinkage



Fig. 2. Radiograph of Class III restorations.
A is made from a commercial product,
B is made from the experimental material.

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